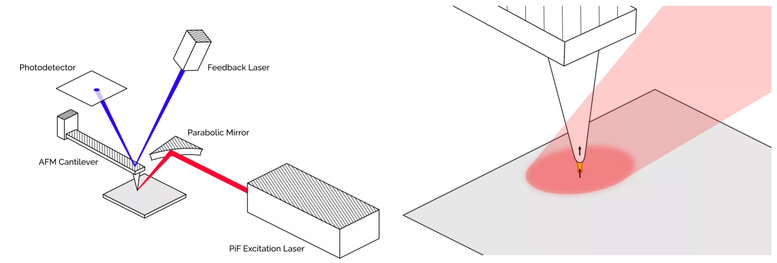
Robust, Quantitative IR-AFM

Why more robust, quantitative AFM?

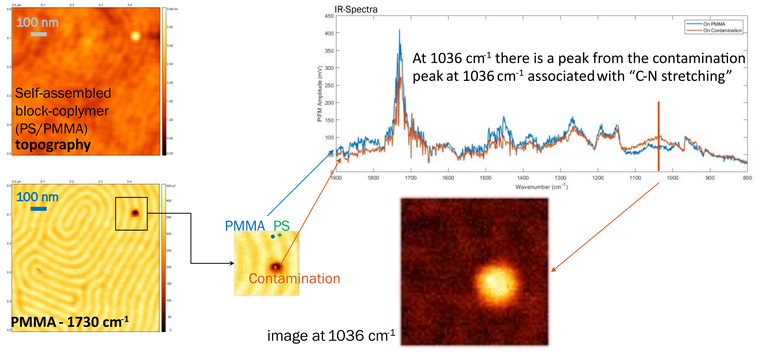
High-throughput (optical) metrology measures necessarily averages over many device structures. It then uses models to interpret results in terms of individual devices. However, modelling the increasing complexity of the structures to the increasingly tight specifications demanded by future processes means these models will contain many parameters and are going to be hard to fit correctly. These considerations lead to a call for multi-modal metrology where different modalities are combined in one analysis to make use of the strong points of each. Here is an opportunity for unconventional metrology techniques to fill in gaps in what conventional metrology can provide. One such unconventional metrology technique is Scanning Probe Microscopy (SPM). It is especially complementary to optical techniques: it provides high resolution, non-destructive measurements of individual device structures with potentially a large range of material properties – we will focus on chemical identification using tip-enhanced IR spectroscopy in this contribution. However, there are some unique caveats with SPM based techniques: they rely on inherently variable - because man-made - objects (the tips) which are sensing through highly non-linear interactions the complex device structures. These two aspects make it difficult to interpret quantitatively signal levels, or even to guarantee a certain sensitivity. In its typical lab-environment application this is not a problem, and people know routinely to extract useful information at the limits of theoretically attainable sensitivity, although sometimes operating a SPM is considered more a handicraft than a science. However, for automated application as an inline metrology tool, it is paramount that sensitivity and quantitative interpretation are guaranteed and understood. Here we report on our steps towards this goal.

IR-AFM Measurement principle



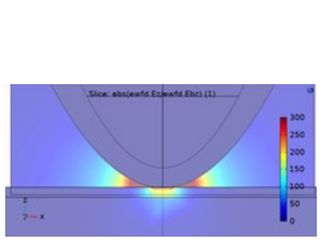
IR-AFM (or PiFM - Photo-induced Force Microscopy) is a technique based on Atomic Force Microscopy. In addition to regular tapping mode topography imaging, a metal coated tip and a pulsed laser laser enable contrast based on IR absorption and subsequent thermal expansion or based on light-induced dipoles, depending on material properties. Resolution is basically set by the tip shape, and is typically reported as below 10 nm. Sensitivity can be sub-monolayer.
Image source: Molecularvista.com/technology/pifm-and-pif-ir/scientific-principles/

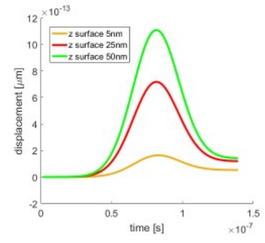
IR-AFM Capabilities

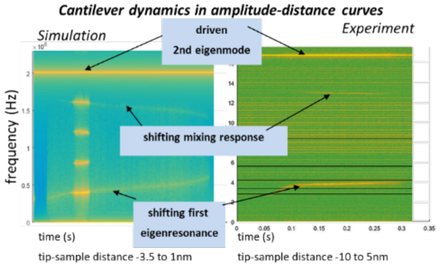


IR-AFM can map different chemical components down to a lateral size of less than 10 nm. An example of this capability is shown on the left where one of the two phases of a self-assembled block copolymer is highlighted using its specific IR absorption wavelength. However, it is also possible to park the tip on a specific location and scan the IR wavelength to obtain a spectrum specific to that location on the sample. Such a localized IR spectrum is shown on the top right, highlighting the possibility to specifically characterize the small contaminating particle on top of the self assembled structure. As further demonstration, the bottom right image is taken at the wavelength specific to the contamination.

IR-AFM Modelling

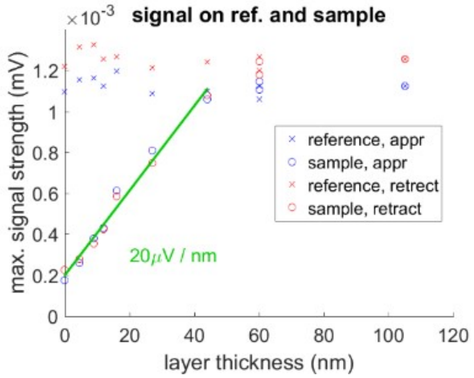






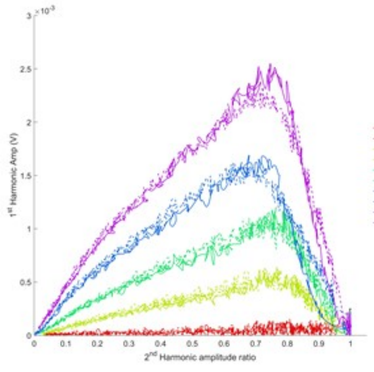
Robust, quantitative AFM starts with a detailed understanding of the contrast formation. To build up that knowledge, we perform simulations of all physics involved in the measurement. Here shown are examples of first of all, on the left, Finite Element Analysis of the ElectroMagnetic Field around the tip-sample contact. Clearly visible here is th strongly enhanced field between tip and sample due to the antenna effect of the metal coated tip and the multiple reflections between tip and sample. This field distribution can be used to calculate the force between tip and sample due to optically induced dipoles (center left). Here the force is shown as function of distance between tip and sample for two different sample configurations. The field distribution can also be used to calculate the heat deposited into the sample by the IR light. Using a time-dependent intensity then allows to calculate the thermal expansion (center right), here shown for three different top layer thicknesses. Depending on material properties, either the thermal expansion or the optically induced dipoles will dominate contrast; for the materials simulated here (Silicon Oxide or PMMA), thermal expansion dominates the contrast. Finally, the material response is measured through the resulting cantilever motion in a complex measurement scheme involving frequency mixing. The highly non-linear tip-sample interaction creates a complex response, as can be seen in both simulations and experiments (right).

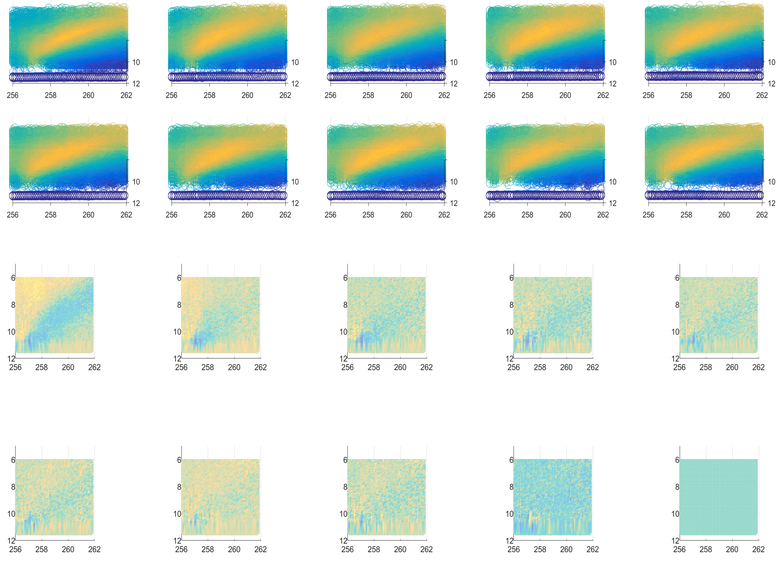
IR-AFM Reproducibility



We approached reproducibility experimentally from different angles. On the left is a set of measurements (each symbol is a measurement) where each measurement was carefully set up for reproducibility. The graph shows signal level as function of thickness of the top layer of material. After each sample, also a measurement on a reference sample (thickest layer) was performed to assess reproducibility, indicated by the crosses here. While there is some offset between signal level as measured on approach to or retract from the sample, the results show a nice reproducibility.

On the right is a graph showing signal level as, basically, function of setpoint for various laser intensities (0, 5, 10, 15 and 20%). As can be seen, the maximum signal level depends linearly on laser intensity, although best setpoint varies slightly depending on this parameter.





On the left is a more complex set of measurements investigating sensitivity and reproducibility according to a few different parameters. X axis of each plot shows the demodulation (and IR pulse) frequency for the IR absorption signal (around the 1st mode eigenfrequency), vertical axis shows the topography feedback (2nd mode) amplitude reduction ratio (from 12 to 6 mV or about 50%). The colour scale indicates the cantilever response amplitude, indicative of IR-absorption in the measurement. When away from the sample, with close to 12mV 2nd mode amplitude, there is no IR absorption signal (dark blue). When close the sample there is a small jump-to-contact, indicated by the lack of data between 12 and 10mV. With futher amplitude reduction, the IR absorption signal increases, and then decreases again, depending on the demodulation frequency. We see here the shift upward of the 1st mode cantilever resonance upon increased interaction stiffness. We also see that the quality factor of the resonance and / or the effectiveness of the contrast mechanism decrease when the contact becomes stiffer.

The two top rows show a set of such measurements taken over a two hour period. The bottom two rows show the difference of each plot with the last one measured. From these results we can conclude that overall, the measurement is quite stable. The first measurement shows a slightly larger Q factor, while the one-but last measurement shows a slight overall difference in amplitude level, but otherwise changes between plots are around the noise level of the measurements.